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Determination of Sulfur Dioxide in Frozen Peeled Shrimp and Dehydrated Pineapple

Masahiro IWAIDA, Yoshio KANEDA, Yoshio ITO and Yasuhide TONOGAI

Modified Monnier-Williams method is widely applied for the determination of residual sulfite in foods. From our experiment it became clear that the recovery of sulfite by this method was rather low, being about 80% with standard sodium bisulfite solution and around 50% with frozen peeled shrimp and dehydrated pineapple. Colorimetric determination of sulfite was tried by us by the adoption of mercury tetrachloride-*p*-rosaniline color developing system. The recoveries of added sulfite to frozen peeled shrimp and dehydrated pineapple were found to be as high as 90%. Besides, this method is rather simple and can be applied for the determination of microquantity (less than 100 ppm) of residual sulfite in foods. Thirteen samples of imported frozen peeled shrimp and 5 samples of dehydrated pineapple were subjected for the determination of residual sulfite by use of this colorimetric procedure. From the results it was confirmed that the residual sulfite levels of all the samples were within the domestic standards (less than 0.1 g/kg for peeled shrimp and less than 0.5 g/kg for dehydrated pineapple when the residual sulfite is expressed as SO_2).

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Background

Shrimps not frozen after the catch turn rapidly black and fetch lower commercial values. It is known that blackening is prevented by soaking the shrimps in a solution of sulfite. When the regulation standards for food, additives and the like were partially amended on April 28, 1973 through ordinance No. 98 of the Ministry of Health and Welfare the use of sulfites such as sodium hydrogensulfite for peeled shrimps was allowed to the extent that the sulfur dioxide residue was 0.1 g/kg, and the regulation was immediately implemented. It became therefore necessary to establish a method of measuring the sulfur dioxide contents in peeled shrimps. As for the volume of sulfurous

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acid bleaching agent used for dehydrated pineapple, a sulfur dioxide residue of less than 0.5 g/kg is allowed. However, the quantitative determination values obtained so far by the distillation method have problems of scattering. We examined the residual sulfur dioxide (sulfurous acid) in imported frozen peeled shrimps and in imported dehydrated pineapple and present here our results together with some comments.

1. Examination of the modified Monnier-Williams method

In the health testing methods[1)], the method of testing sulfurous acid in foodstuffs is to first to oxidize the distillate with hydrogen peroxide by the distillation method and then to use the alkali titration method (modified Monnier-Williams method). However, when we examined this method, we found that the recovery rate of sodium hydrogensulfite alone was at most 80% and that the reproducibility was poor. When actually adding the substance to samples, the recovery rate was still lower. There are also differences in yield rate depending on the type of sample. We were therefore of the opinion that exact determinations of sulfurous acid were not practically possible with this method.

2. Examination of the p-rosaniline colorimetric method

In 1973, Nakamura et al.[1)] of the Kanagawa Prefectural Health Research Center reported having obtained good results when applying the colorimetric method using a p-rosaniline-formaldehyde solution to the

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quantitative determination of residual sulfurous acid in frozen peeled shrimps. We decided to examine this method. We summarize the procedure as follows.

[Reagents]

Mercury tetrachloride solution: We dissolved 27.2 g of mercuric chloride and 11.7 g of NaCl in a 5% glycerol solution, brought to 1000 mL and added 0.03 g of sodium azide. We added 3000 mL of distilled water to this 1000 mL solution and stirred.

p-rosaniline-formaldehyde solution: We dissolved 0.2 g of p-rosaniline hydrochloride in 1000 mL of water. We collected 20 mL thereof, added 6 mL of hydrochloric acid, brought to 100 mL with water. We also diluted newly standardized formaldehyde with water so as to have a 0.2% solution and prepared a 100 mL mixture.

[Determination method]: After adding 70 mL of mercury tetrachloride solution to 10 g of sample and homogenizing for 2 minutes, we added a saturated corrosive sublimate solution and brought the total volume to 100 mL. After shaking vigorously, we separated by centrifugation, filtered the supernatant with a Toyo filter paper No. 5C and used 1 mL of filtrate (or a diluted solution of the same) as sample solution. We added 1 mL of p-rosaniline-formaldehyde solution, brought to 10 mL with the mercuric tetrachloride solution and left standing for 35 min. Comparing with blank test samples, we measured with a 560 mμ wavelength the absorbance of the colored solution obtained.

[Comments] The mercury tetrachloride solution prepared by the $HgCl_2 + 2NaCl \rightarrow Na_2[HgCl_4]$ reaction from the corrosive sublimate and sodium chloride reacts as follows with the sulfur dioxide and generates mercury complex salts. As it reacts with the p-rosaniline-formaldehyde solution it displays a particular color[3]. p. 93

$$Na_2[HgCl_4] + 2SO_2 + 2H_2O \rightarrow Na_2[Hg(SO_3)_2] + 4HCl$$

[Results of the test]

When drawing the standard calibration curve of SO_2 from sodium hydrogensulfite, we obtained rectilinearity between 10 and 50 μg. Table 2 shows the results of the addition-recovery tests with regard to frozen peeled shrimp and dehydrated pineapple. The recovery rates based on this method exceeded 90% for both the frozen peeled shrimps and dehydrated pineapple. Both rates were clearly superior to those obtained with the modified Monnier-Williams method (see Table 1). As Nakamura et al. also point out, there is a noticeable problem concerning reproducibility. It is however possible to determine by this method residual sulfurous acid of a level below 100 ppm. Moreover, the method is recommended for the rapidity and simplicity of the procedure.

3. Results of the examination of the imported products

The measurement results for the 13 samples of imported peeled shrimp showed a 0.0021 ~ 0.0115 g/kg (2.1 ~ 11.5 ppm) range of SO_2. They were thus within the standard values (less than 0.1 g/kg). The measurement results for the 5 samples of dehydrated pineapple showed very low levels viz. (0.03 and 0.04 g/kg) in 2 instances, and (0.22, 0.25 and 0.28 g/kg) in the 3 others. All results were within the range of standard values (less than 0.5 g/kg).

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Table 1. Recovery of sulfite by the modified Monnier-Williams method

Sample	Recoveries (%) in tripricate		
Sodium bisulfite solution (equivalent to 2.5 mg of SO_2)	61.9	81.4	79.4
2.5 mg of SO_2 was added in the form of sodium bisulfite solution to 25 g of frozen peeled shrimp	42.7	37.5	46.2
2.5 mg of SO_2 was added in the form of sodium bisulfite solution to 25 g of dehydrated pineapple	49.7	52.2	60.1

Table 2. Recovery of sulfite by the colorimetric method by use of *p*-rosaniline

Sample	Recoveries (%) in triplicate		
Sodium bisulfite solution (equivalent to 250 μg of SO_2)	98.2	99.5	96.1
250 μg of SO_2 was added in the form of sodium bisulfite solution to 10 g of frozen peeled shrimp	93.9	98.3	102.4
250 μg of SO_2 was added in the form of sodium bisulfite solution to 10 g of dehydrated pineapple	90.0	102.5	95.0

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3. Same author, same reference, same publ. as in 1. above, p. 1040 (1973).